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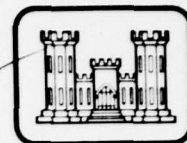
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REPORT 79-18

Insulating and load-supporting properties of sulfur foam for expedient roads in cold regions

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Cover: Applying sulfur foam to test area.
(Photograph by North Smith.)

CRREL Report-79-18

Insulating and load-supporting properties of sulfur foam for expedient roads in cold regions

North Smith and Daniel A. Pazsint

September 1979

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER CRREL Report 79-18	**2. GOVT ACCESSION NO.**	**3. RECIPIENT'S CATALOG NUMBER**
4. TITLE *(and Subtitle)* INSULATING AND LOAD-SUPPORTING PROPERTIES OF SULFUR FOAM FOR EXPEDIENT ROADS IN COLD REGIONS		**5. TYPE OF REPORT & PERIOD COVERED**
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) North Smith and Daniel A. Pazsint		**8. CONTRACT OR GRANT NUMBER(s)**
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Army Cold Regions Research and Engineering Laboratory Hanover, New Hampshire 03755		**10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS** DA Project 4A062112A894 Task 02, Work Unit 001
11. CONTROLLING OFFICE NAME AND ADDRESS Directorate of Military Programs Office, Chief of Engineers Washington, D.C. 20314		**12. REPORT DATE** September 1979
		13. NUMBER OF PAGES 26
14. MONITORING AGENCY NAME & ADDRESS*(if different from Controlling Office)*		**15. SECURITY CLASS. (of this report)** Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*
Cold regions engineering
Expedient construction
Foam
Sulfur
Unsurfaced roads

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Temperatures of the subgrade and of sulfur foam insulation test sections in an expedient road were monitored with thermocouples to document freezing and thawing conditions. Vehicular trafficking was conducted on a limited basis to determine the load supporting capabilities of the foam. The sulfur foam, placed directly under a prefabricated surface mat, was found to be unsuitable for use as an expedient thermal insulation and traffic load supporting material primarily because of its low tensile strength and high brittleness. The insulating value of sulfur foam produced by the batch process in the field was about one-half that of extruded polystyrene, meaning double the thickness for equal protection against thaw.

PREFACE

This report was prepared by North Smith, Research Civil Engineer, Geotechnical Research Branch, Experimental Engineering Division, U.S. Army Cold Regions Research and Engineering Laboratory, and Daniel Pazsint, Chemical Engineer, formerly of CRREL. The study was conducted as part of DA Project 4A062112A894, *Engineering in Cold Environments*, Task 02, *Engineering Design Criteria*, Work Unit 001, *Expedient Roads, Airfields and Heliports in Cold Regions*.

The authors wish to express their appreciation for technical review of this report by Dr. Richard Berg and William Quinn and for the support of the personnel of the CRREL Alaskan Projects Office.

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CONVERSION FACTORS: U.S. CUSTOMARY TO METRIC (SI) UNITS OF MEASUREMENT

These conversion factors include all the significant digits given in the conversion tables in the ASTM *Metric Practice Guide* (E 380), which has been approved for use by the Department of Defense. Converted values should be rounded to have the same precision as the original (see E 380).

Multiply	By	To obtain
inch	25.4*	millimeter
foot	0.3048*	meter
pound-mass	0.4535924	kilogram
ton	907.1847	kilogram
kip	4448.222	newton
pound-force/in.²	6984.757	pascal
pound-force/ft³	16.01846	kilogram/meter³
Btu in./hr ft² °F	0.1442279	watt/meter kelvin
degrees Fahrenheit	$t_k = (t_{o_F} + 459.67)/1.8$	kelvins
degrees Celsius	$t_k = t_{o_C} + 273.15$	kelvins

*Exact

INSULATING AND LOAD-SUPPORTING PROPERTIES OF SULFUR FOAM FOR EXPEDIENT ROADS IN COLD REGIONS

North Smith and Daniel Pazsint

INTRODUCTION

The thawing of ice-rich fine-grained soils in the cold regions of the world creates severe structural support and environmental problems in roads and airfields. In permafrost regions, insulating materials have been used to maintain the frozen state of the substrate, while in seasonal frost areas insulation is used to prevent freezing of the substrate. Insulations, both natural and synthetic, are rather expensive and costly to use; therefore, new candidates are being developed constantly.

This report describes the construction and the evaluation of two expedient road test sections of foamed sulfur covered with expedient surfacing. The investigation was conducted in conjunction with the Southwest Research Institute (SWRI) of San Antonio, Texas, where the sulfur foam concept was developed.

SITE PREPARATION

During the summer of 1970 a site was laid out for two sulfur foam sections (d and e, Fig. 1) in an undisturbed area at the CRREL Farmers Loop Road Facility near Fairbanks, Alaska, using the original centerline of the Expedient Roads Test Sections. In the early fall of 1970 part of the area (approximately 20×20 ft) was cleared of all trees and underbrush leaving the natural vegetation mat intact. The vegetative growth consisted largely of small spruce and willow trees, low cranberry bushes and moss. This area extended from approximately station 2+25 of an adjoining polyurethane test section to station 2+45 (see Fig. 2). No soil testing was done on this area, which generally is not appreciably different from that tested for the polyurethane section.[4] Depths to the top of the permafrost were determined for the area by using an engineering level and a steel probing rod when construction started on 23 September 1970. A 15-ft-square section extending from station 2+27.5 to station 2+42.5 was staked out for the foam application in October 1970. This first sulfur foam pad and subgrade section (d) was instrumented for temperature measurements with three thermocouple assemblies having eight points each. These were installed, as shown in Figure 3, by pouring a slurry of Fairbanks silt (which later refroze) around the positioned points and lead wires. Table I gives the point locations of all thermocouples.

A second 15-ft-square sulfur foam pad (section e) was poured in May 1971, adjacent to the first one, between the expedient road stations 2+42.5 and 2+57.5 (see Fig. 1 and 2). The area was stripped to frozen ground with a front-end loader and bulldozer after being cleared of trees and brush. Frozen soil cores taken from the subgrade thermocouple installation holes were tested for moisture content and dry densities (see Table II). This pad was also instrumented with three thermocouple assemblies of eight points each. These assemblies were also installed with a silt-slurry at the locations shown in Figure 3.
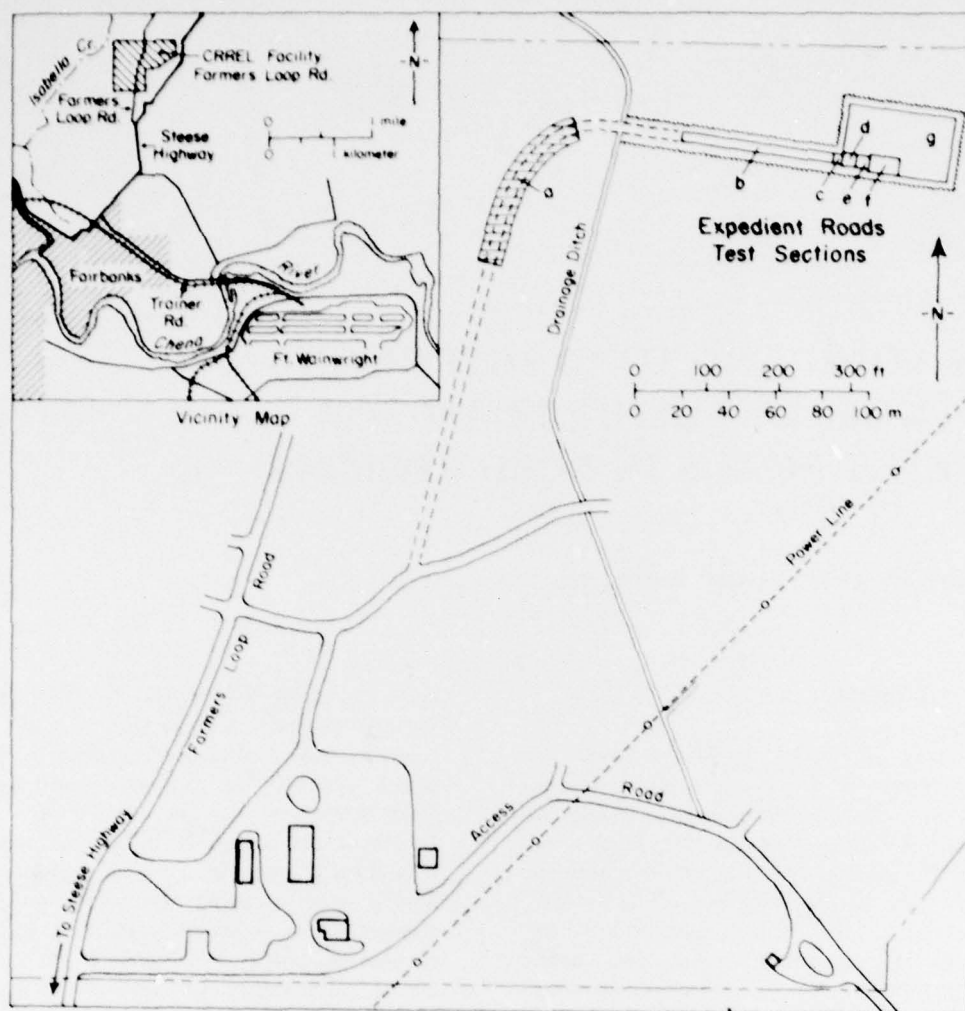
Figure 1. General plan of the CRREL Farmers Loop Road Facility.
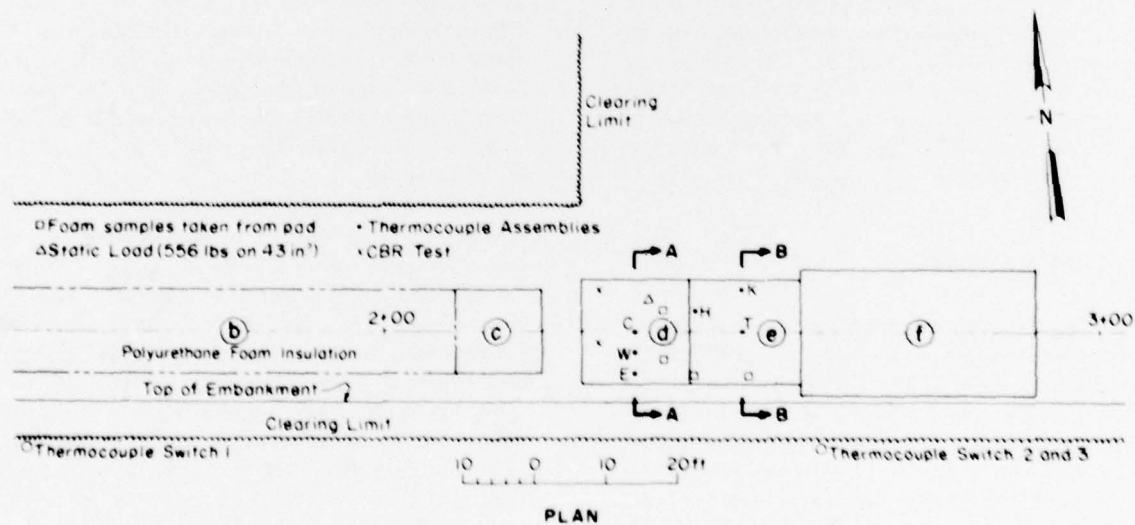
Figure 2. Site plan of sulfur foam sections and thermocouple assemblies.
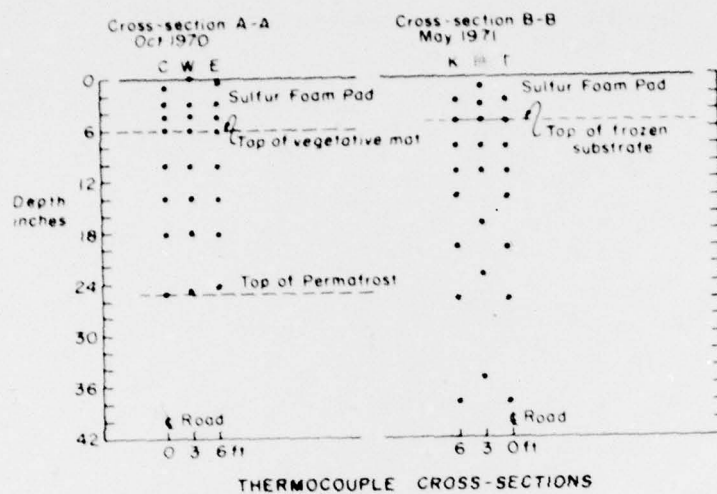
Figure 3. Thermocouple assembly profiles.

Table I. Thermocouple point locations.*

Switch position	Depth below foam surface (in.)	Switch position	Depth below foam surface (in.)	Switch position	Depth below foam surface (in.)
October 1970 sulfur foam pad (section d)					
C-8	1.0	W-8	0	E-8	0.5
C-7	3.0	W-7	3.0	E-7	3.0
C-6	4.5	W-6	4.5	E-6	4.5
C-5†	6.0	W-5	6.0	E-5	6.0
C-4	10.0	W-4	10.0	E-4	10.0
C-3	14.0	W-3	14.0	E-3	14.0
C-2	18.0	W-2	18.0	E-2	18.0
C-1**	25.0	W-1	24.7	E-1	24.2
May 1971 sulfur foam pad (section e)					
K-1	2.5	T-1	2.5	H-1	1.0
K-2	5.0	T-2	5.0	H-2	3.5
K-3	8.0	T-3	8.0	H-3	5.0
K-4	11.0	T-4	11.0	H-4	8.0
K-5	14.0	T-5	14.0	H-5	11.0
K-6	20.0	T-6	20.0	H-6	17.0
K-7	26.0	T-7	26.0	H-7	23.0
K-8	38.0	T-8	38.0	H-8	35.0

*Refer to Figure 3
†Top of vegetation mat
**Top of permafrost

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Table II. Properties of subgrade soil beneath sulfur foam pad. [*]

Material	Location (station)	Depth (ft)	Moisture content (%)	Dry density (lb/ft³)
Organic silt	2 + 42.5	0 to 1.0	39.4	
	3 ft left of ℄	1.0 to 1.45	98.6	38.0
		1.45 to 2.05	98.6	
		2.05 to 2.65	65.2	
Organic silt	2 + 48.5, ℄	0 to 0.65	47.3	
		0.65 to 1.2	73.4	
		1.2 to 1.85	119.5	36.3
		1.85 to 2.55	72.9	
Organic silt	2 + 48.5	0 to 0.55	71.8	52.0
	6 ft left of ℄	0.55 to 1.1	56.8	62.7
		1.1 to 1.65	135.0	
		1.65 to 2.2	94.5	
		2.2 to 2.85	57.8	

[*]Under May 1971 sulfur foam pad

SULFUR FOAM APPLICATION

Installation of the first sulfur foam test section (d) was started late on 30 September 1970 and continued during the first two days of October 1970. The equipment setup consisted of SWRI's steam-jacketed reactor vessel, and CRREL's accompanying steam boiler, electric generator, and air compressor.

The sulfur was delivered in barrels in lump form from Taylor, B.C., Canada, where it was recovered from sour natural gas. Other chemicals used to produce the foam were shipped from San Antonio, Texas.

The sulfur foam mixture was of the same formulation as reported in CRREL Technical Report 227.[2] Seven batches of foam prepared to construct this test pad had the following composition:

Component	Weight (lb)
Sulfur	300.0
Talc	30.0
P_2S_5 (phosphorus pentasulfide)	15.0
N_3PO_4 (phosphoric acid)	15.0
1,5-cyclooctadiene	9.0
TCP (tricresyl phosphate)	0.75

Although most of the later batches used sulfur premelted in a make-shift, steam-heated barrel, the process was intended to be conducted by melting the sulfur in the reactor. This premelting of the sulfur reduced the preparation time per batch by as much as an hour. As the sulfur melted and its temperature stabilized at 290°F (the limit of the reactor steam jacket) the talc, P_2S_5, TCP and 1,5-cyclooctadiene were added and mixed by stirring during the reaction. After mixing for approximately 30 minutes, the phosphoric acid was added and mixing continued.[1,2] The reaction product was then forced out of the reactor through hoses by air pressurization of the reactor vessel. The foam was applied in 1- to 2-in. layers as it oozed from the hose with a consistency of pancake batter (Fig. 4 and cover). The surface of the foam was tacky for about an hour and the test pad could not be walked on without damage until three to four hours after pouring.

The test pad was poured in two or three layers to obtain a relatively even surface at the required thickness. About half of each prepared batch was applied to each half of the pad, front and rear (west and east), and a final batch was used as a leveling layer and to make test samples. Three wooden test sample boxes (Fig. 5) were used as molds and shipping containers for the four samples. These were extensively tested and compared with sulfur foam samples taken directly from the pad.

Section e was poured on 15 May 1971 when the ground surface was starting to thaw. The

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Figure 4. Finish application of foam, showing consistency and application technique, May 1971.
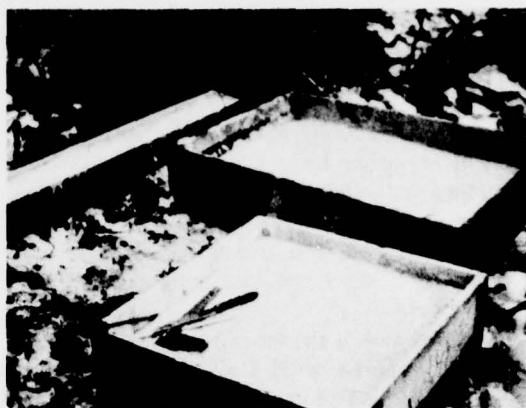

Figure 5. Wooden boxes with poured sulfur foam samples, October 1970.

Figure 6. Laying fiberglass mat on frozen substrate, May 1971.

Figure 7. Applying LP-3 modified sulfur foam onto fiberglass mat, May 1971.

Figure 8. Foam application on frozen substrate of rear half of pad, May 1971.

Figure 9. Rolling molten sulfur on fiberglass webbing (finished surface), May 1971.

reactor equipment used in the October 1970 foaming had been winterized and stored on the site for use on this pad. The formulation differed from the previous one by the addition of a liquid plasticizer, Thiokol LP-3, to reduce the brittle nature of the cured foam. This polysulfided ethylene compound was added to the first batch at a 2½% by weight ratio. Since the first batch of foam overheated, presumably due to the addition of the LP-3, the amount of additive was reduced to 1½% by weight for the remaining five batches.

The front half of the frozen subgrade test pad area was covered with 1½-in-thick fiberglass matting (Fig. 6). The matting extended over the entire width of the 15-ft-wide pad from station 2+42.5 to station 2+50 and was expected to provide added tensile strength to the pad. Two batches of foam were poured onto the fiberglass matting (Fig. 7) and two more batches were poured on the rear half of the pad (Fig. 8). The fifth batch was used to fill in some low spots located generally in the middle and left rear quadrant of the pad. The hardened pad was then covered in a crisscross pattern with a thin fiberglass webbing. Molten sulfur was rolled onto this webbing to bond it to the foam surface (Fig. 9) in an attempt by SWRI personnel to provide a more durable foam pad surface.

SULFUR FOAMING EQUIPMENT

The reactor employed by SWRI personnel for the installation of these sulfur foam test sections was a converted, steam jacketed pressure vessel made of mild steel and operated at 80-psi steam pressure. An electric motor mounted on top of the reactor was used to drive a set of mixing paddles inside the vessel. Compressed air was used to expel the molten foam through insulated hoses. This batch mixing reactor was charged with the foam components, except the phosphoric acid, through a hatch in the top (Fig. 10). The phosphoric acid was added through a valved inlet line at the top of the vessel with the use of a funnel. A pipe connection on the reactor was available for applying pressurized steam directly to the foam discharge lines to clear out solidifying sulfur foam after each application.

The reactor performed well with no mechanical failures. The length of time required to prepare a 300-lb batch was probably in the 1½- to 2- hour range, although batches could conceivably be cycled through the reactor in about an hour with premelted sulfur available. There were problems in producing foams of uniform quality in different batches, as might be expected in a batch-type chemical process.

ENVIRONMENTAL ASPECTS

Ambient air temperatures during the construction of the sulfur pad in September-October 1970 were moderately cool, with highs of 25° to 35°F and lows of 13° to 25°F. Occasional light snowfall during the test accumulated to a 1- to 2-in. ground cover and actual foaming took place on top of a 1-in. snow-covered vegetative mat (Fig. 11). The subgrade surface temperature

Figure 10. Open loading hatch, stirrer drive and air pressure line on SWRI reactor.

Figure 11. Applying sulfur foam on snow-covered vegetative mat, October 1970.

was 30°F during the initial application of foam. The operation of all equipment used in the construction process was not adversely affected by these weather conditions. Obviously, some temperature drop was expected in the foam after being expelled from the reactor at 290°F while passing through 15-20 ft of hose. Considering the above heat loss, the temperature of the foam at the nozzle was probably in the 250-260°F range as evidenced by the rapid solidification on the ground (elemental sulfur solidifies at approximately 234°F). The cold snow layer on the substrate caused the foam to harden quickly and form a dense bottom layer in the 60-80 lb/ft³ density range. The report[1] by SWRI on this project has some additional data indicating that similar results occurred when sulfur foam was applied to dry ice.

The completed pad showed signs of surface cracking within a day or two after completion. The thinner edges of the pad separated from the main mass during this time because of shrinkage. Thermocouple readings taken before, during and after pouring the foam showed that the subgrade surface reached a maximum of 37°F while the temperature 4 in. below the subgrade surface and deeper remained unchanged at essentially 32°F.

Weather conditions during the May 1971 sulfur foaming were more moderate than in the fall with highs in the 40-60°F range and lows in the 30's. Most of the snow had melted by the time the stripping operation for the pad was started. Due to the warmer temperatures involved, some portions of the subgrade had a ½- to ¾-in.-thick melted surface layer of organic silt. The sulfur foam was applied on this stripped and partially thawed substrate. The high moisture content of the surface had no immediate noticeable adverse effects on the application of the foam. Also, no difficulty was encountered during the application of the melted sulfur surface treatment to the warm foam pad. Although no thermocouple readings were taken during foaming, it was determined that the thaw line did\not reach the 3-in. depth for 10 days.

The reactor and other equipment operated successfully in this environment, although some moisture that had collected in the reactor during winter storage at the site had to be evaporated.

TRAFFIC TESTING AND
TEST PAD OBSERVATIONS

The weekly readings of the thermocouples installed within and below the test pads were used to indicate the rate and depth of thawing and freezing of the active soil layer. Observations were discontinued during the winter when the active layer became refrozen to the depth of the deepest thermocouple. Figures 12 and 13 show temperature curves in the insulation and subgrade for the October 1970 and May 1971 pads, respectively. Figures 14 and 15 show surface and permafrost elevations and Table III
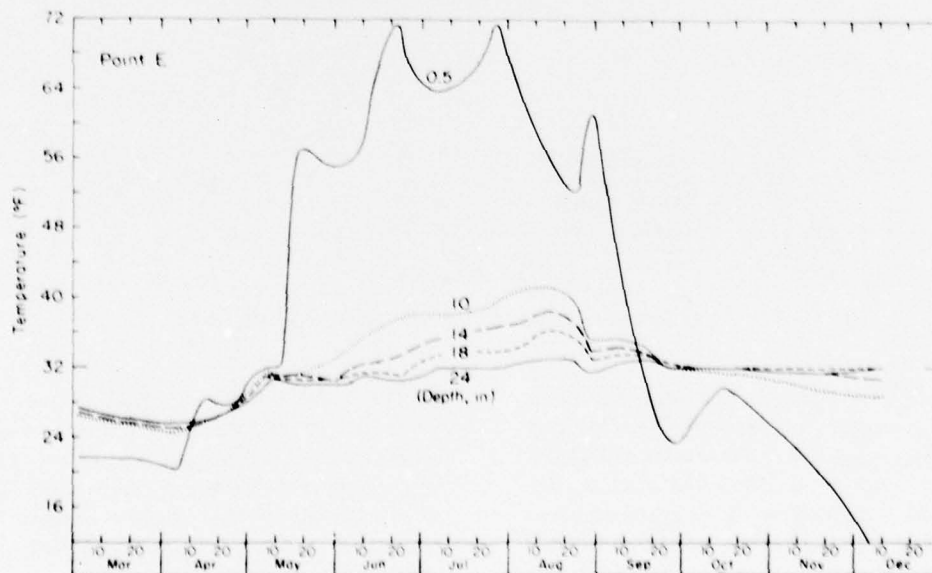
Figure 12. Thermocouple assembly readings in section d during 1971.
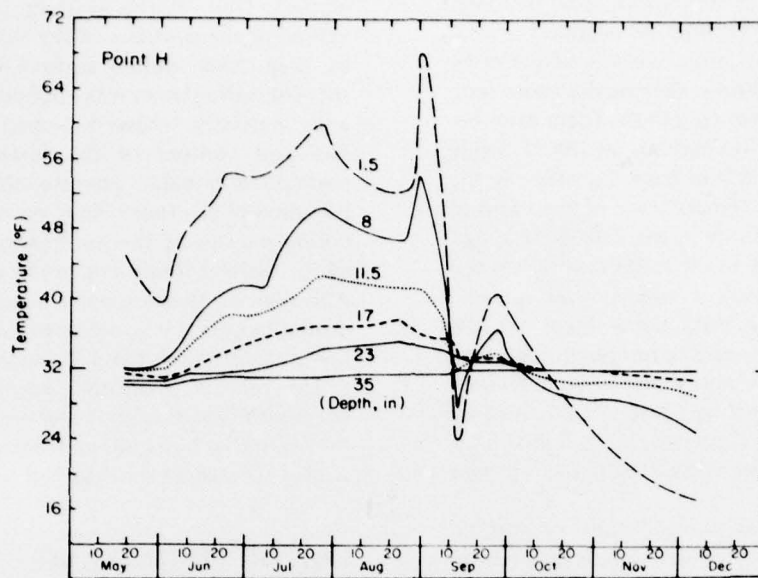
Figure 13. Thermocouple assembly readings in section e during 1971.
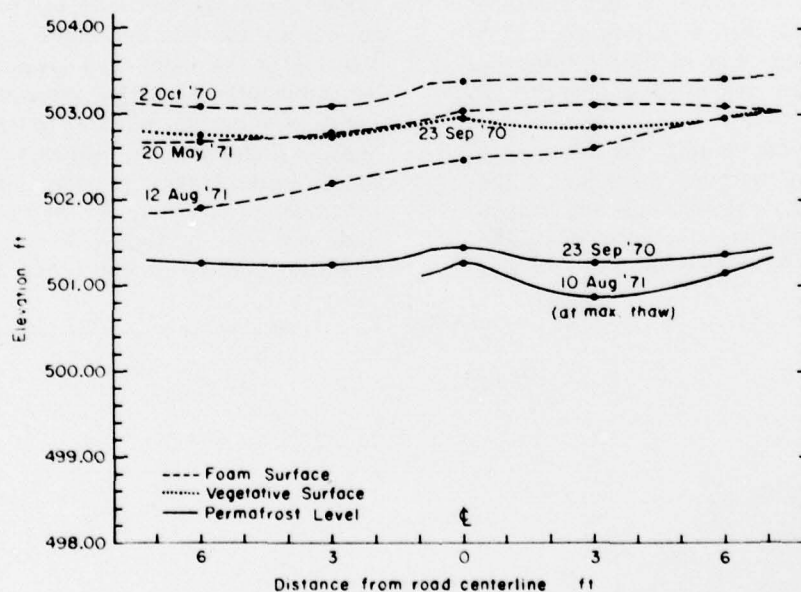
Figure 14. Surface and permafrost elevations of section d.

gives the maximum thaw depths under the pads during 1971. Figure 16 presents the freezing and thawing indices for the 1970 winter and 1971 summer, and Figure 17 shows the plan of the thermocouple assembly in an undisturbed area. Assembly 13 in Figure 17 is a control thermocouple assembly installed at an undisturbed site near the Expedient Road Test Sections.[5]

The natural surface insulating qualities of the area and those provided by sulfur foam can be compared by referring to Figures 14, 15 and 18. Figures 14 and 15 show maximum thaw depths of 2 to 3 ft beneath the sulfur foam pads. Figure 18 shows the ground temperatures at this site during 1971 and the maximum thaw depth for the year of 3.0 ft which occurred in August. During
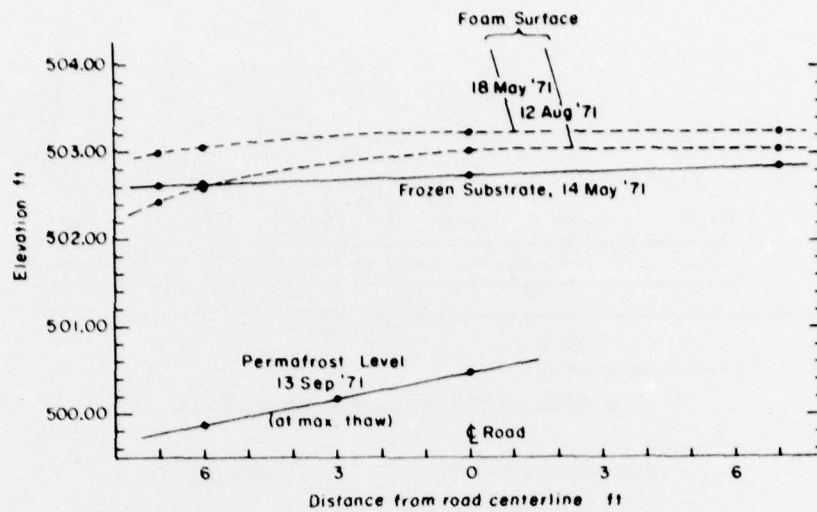
Figure 15. Surface and permafrost elevations of section e.
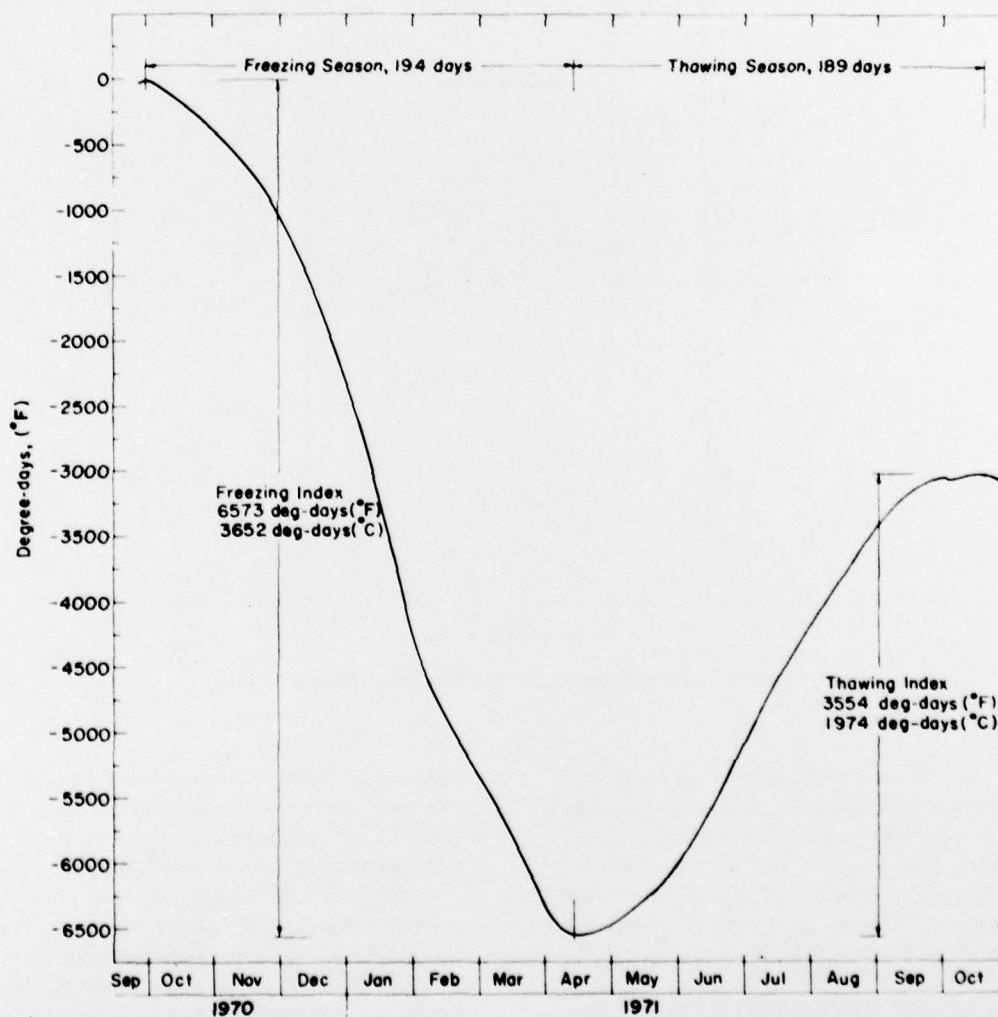
Figure 16. Air freezing and thawing indices for Fairbanks, Alaska.

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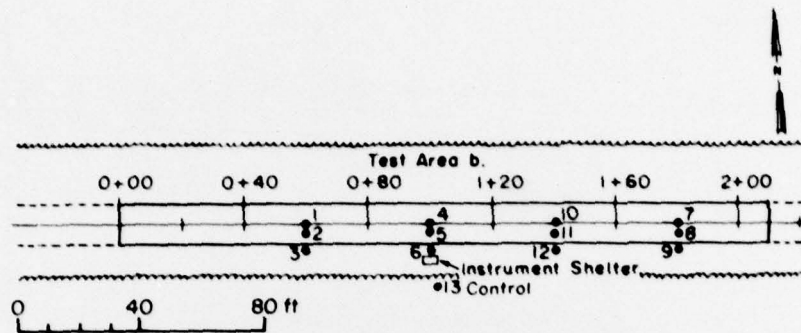
a. Plan of Thermocouple Assemblies
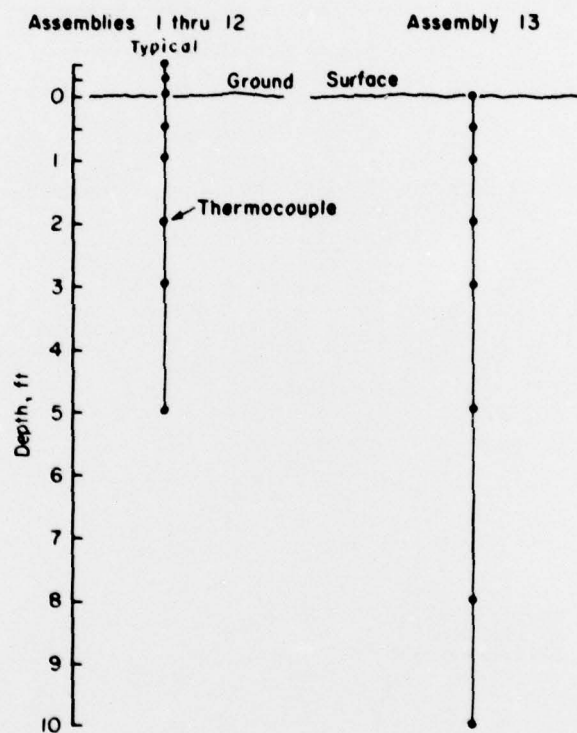
b. Profiles

Figure 17. Thermocouple assembly in undisturbed area.

the early part of May 1971, the sulfur foam pad was subjected to foot traffic and snow clearing equipment operations. Those operations caused some minor compressive and flexural failures which allowed infiltration of meltwater during spring thawing. It later adversely affected the performance of the test sections under vehicular traffic.

A static load test (location shown in Fig. 2) on the October 1970 pad for the 1970-1971 winter resulted in some compressive failure although the test was accidentally tipped over late in the winter by snowmobile traffic. Additional damage was caused by the falling weights.

Some foot traffic also occurred on the newly installed May 1971 sulfur foam pad before MOMAT (a fiberglass-reinforced plastic mat) was placed over both pads and the vehicular traffic was started on 21 May 1971. At that time a 20-ton wrecker and a tractor trailer rig were
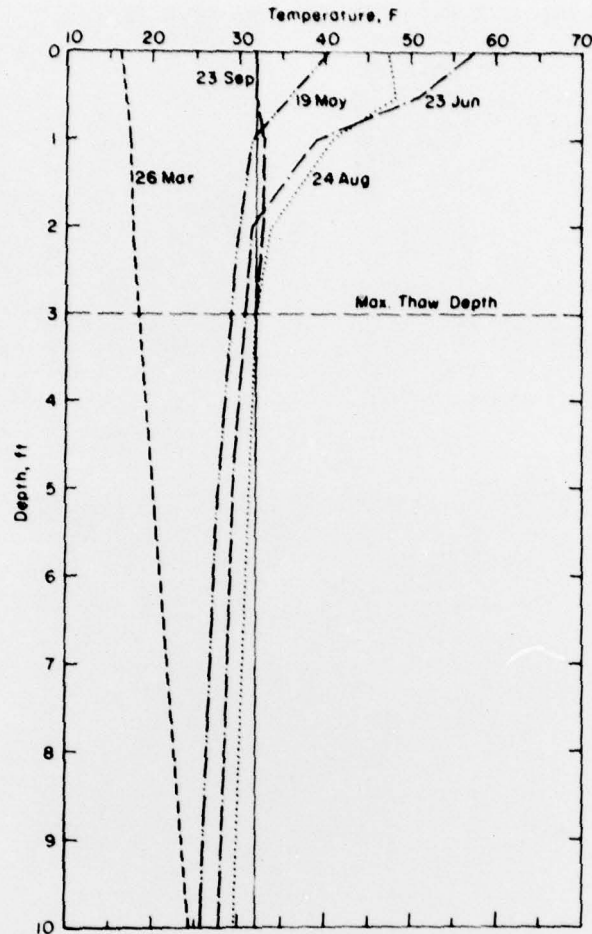
Figure 18. Temperatures in undisturbed area during 1971.

Table III. Thaw factors and maximum thaw for sulfur foam in Fairbanks.

| | Foam thickness | | Maximum thaw depth* | | |
Foam pad	Range (in.)	Avg (in.)	Date	Location (station)	Depth† (in.)
Oct 1970 Sulfur	2.0-6.7	4.2	10 Aug 71	2 + 35 6 ft right of ₵	20.6
Oct 1970 Sulfur			10 Aug 71	2 + 35 3 ft right of ₵	23.5
Oct 1970 Sulfur			10 Aug 71	2 + 35, ₵	20.7
May 1971 Sulfur (LP-3)	2.6-8.5	4.9	13 Sept 71	2 + 43.5 3 ft left of ₵	30.0
May 1971 Sulfur (P-3)			13 Sept 71	2 + 50 6 ft left of ₵	33.0
May 1971 Sulfur (LP-3)			13 Sept 71	2 + 50, ₵	27.0

*Obtained during 1971 from thermocouples beneath sulfur pads.
†Depth below original surface elevation.

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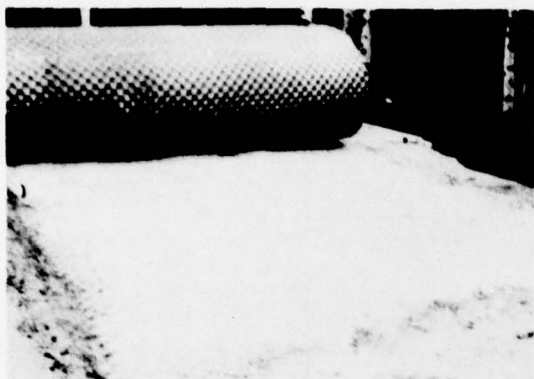
Figure 19. Section e after light traffic on MOMAT surface.
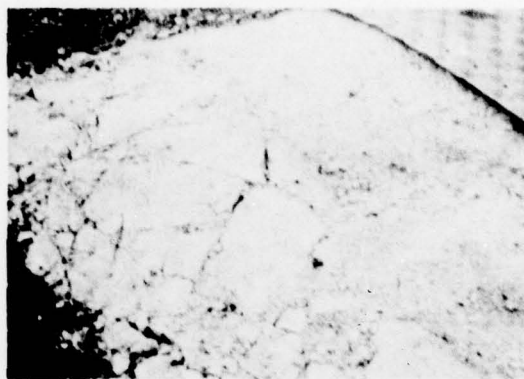
Figure 20. Condition of section d after light traffic on MOMAT surface, June 1971.

Figure 21. Condition of section e after light traffic on MOMAT surface, June 1971.

driven over the sections causing moderate damage, the October 1970 pad received more damage due to its weakened condition from the spring melt and the highly flexible underlying vegetative mat. Some additional sedan and carry-all type traffic traversed the pads during the remainder of May 1971 and early June 1971. In August 1971 the MOMAT was removed for another use and replaced with XM-18 landing mat. Occasional light traffic use continued along with several trips with the 20-ton wrecker and a loaded test cart used on the adjacent foamed-in-place polyurethane airfield test section. No rigorous trafficking schedule was followed in evaluating the sulfur foam pads. The observations made during and after the 1971 thaw season indicated that, as an expedient mat-covered insulation, the pads provided much less load support than the polyurethane sections

previously and concurrently tested (Fig. 19, 20, 21).

FIELD SAMPLING AND TEMPERATURE READINGS DURING POURING

During the October 1970 foam placement, two samples were cut from the rear section of the pad, as shown in Figure 22, for density measurements and further laboratory testing. These samples had densities in the range of 35 to 67 lb/ft^3. Also, parts of four batches were foamed into separate cardboard containers, and tests on sections of these samples gave densities in the 8 to 18 lb/ft^3 range.

Thermocouple readings were taken during the final stage of foam application in October 1970. The graphs shown in Figures 23 and 24 are

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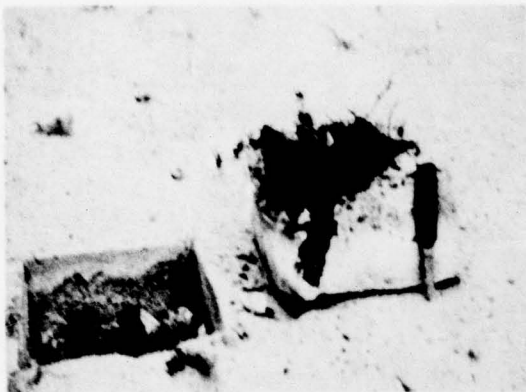
Figure 22. Sulfur foam sample cut from section d.

typical time-temperature curves within and below the October 1970 sulfur foam pad. These readings were taken during the application of the last layer of foam to the pad and during the subsequent cooling period. They show the delayed effect of rising temperatures, due to the insulating qualities of the foam, when molten foam at 250°-260°F was applied to the surface. Likewise, they also show the delayed effect of

heat loss to the 32°F subgrade and the slightly colder ambient air. A close estimate of the thermal conductivity of the foam pad that was poured in October 1970 can be obtained by making calculations on the cooling part of the curves, i.e. the portion that occurs after heat input has ceased, the foam has solidified and the curves have begun to converge into a smooth array. The following timetable indicates the significance of each of the nine sets of readings on these curves.

1 October 1970

0900	Base reading	Only C-8 T/C lead exposed
1210	No reading	Foaming started
1230	Reading	5 minutes after foam covered C-8 T/C lead
1240	Reading	
1305	Reading	
1310	No reading	Thin foam added above T/C assembly C
1340	Reading	5 minutes after maximum surface temperature recorded
1445	Reading	

Figure 23. Cooling curves of section d, thermocouple assembly W.

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Table IV. Properties of sulfur foam.

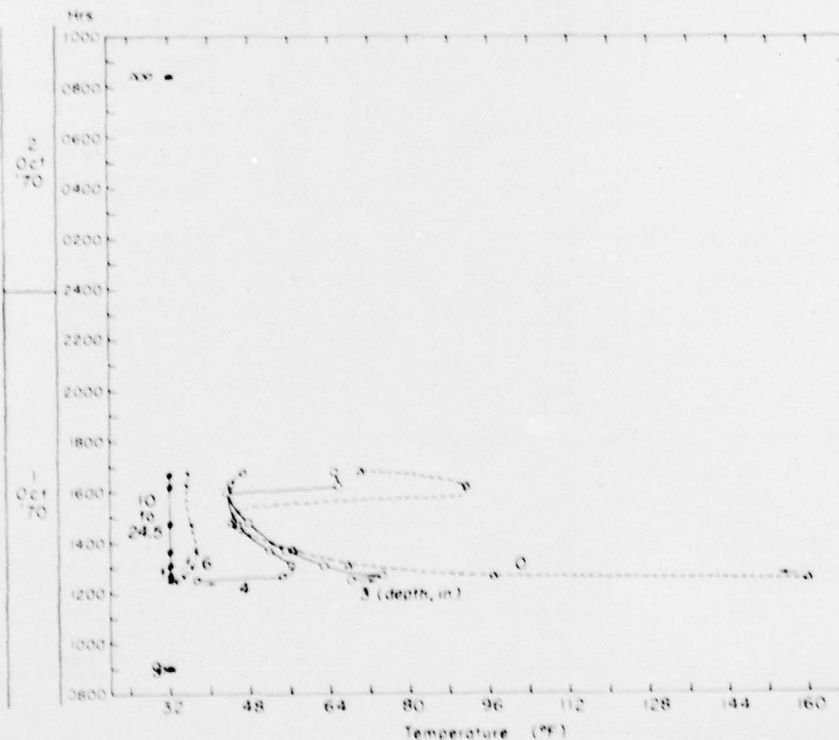| Test	ASTM no.	Value		Density (lb/ft³)	Source of material
Water absorption	D2842	27.3	(volume %)	34.3	Oct 70 foam pad
		25.9		26.3	Oct 70 foamed in box
		22.9		36.7	Oct 70 foam pad
		7.90		22.6	May 71 foam pad
		7.48		22.9	May 71 foam pad
		5.76		21.3	May 71 foam pad
Field CBR		16	(% at 2 in.)	36	Oct 70 foam pad (Sta 2 + 30, 1.5 ft right of ₵)
		12		32	Oct 70 foam pad (Sta 2 + 30, 6 left of ₵)
Compressive strength	D1621	28.0	(lbf/in.²)	12.8	Oct 70 foamed in box
		29.2		19.2	Oct 70 foamed in box
		23.5		19.5	Oct 70 foamed in box
		7.6		20.0	Oct 70 foamed in box
		102.7		20.7	Oct 70 foamed in box
		41.4		21.7	Oct 70 foamed in box
		46.7		22.7	Oct 70 foamed in box
		52.5		23.1	Oct 70 foamed in box
		89.0		23.8	Oct 70 foamed in box
		62.8		24.7	Oct 70 foamed in box
		18.1		24.8	Oct 70 foamed in box
		123.4		34.2	Oct 70 foam pad
		92.3		35.5	Oct 70 foam pad
		91.1		37.5	Oct 70 foam pad
		145.0		38.2	Oct 70 foam pad
		61.0		17.6	May 71 foam pad
		32.3		19.6	May 71 foam pad
		44.6		20.6	May 71 foam pad
		51.2		21.9	May 71 foam pad
		58.0		26.1	May 71 foam pad
		24.5		20.8	Mar 71 SWRI sample
		21.2		21.5*	Mar 71 SWRI sample
		22.4		13.1	Mar 71 SWRI sample
		24.5		13.5†	Mar 71 SWRI sample
Compressive strength (before and after water absorption)**	D1621	47.2	(lbf/in.²)	27.4††	Oct 70 foamed in box
		36.1		31.9***	Oct 70 foamed in box
		44.6		20.0††	May 71 foam pad
		42.3		22.2***	May 71 foam pad
Compressive strength (before and after freeze thaw test)†††	D1621	123.4		34.2††	Oct 70 foam pad
		81.8		48.6***	Oct 70 foam pad
		145.0		38.2††	Oct 70 foam pad
		49.7		51.1***	Oct 70 foam pad
		87.2		21.7††	May 71 foam pad
		92.9		30.9***	May 71 foam pad
		89.1		21.8††	May 71 foam pad
		46.7		34.6***	May 71 foam pad
Freeze-thaw	C291	7	(cycles before failure)	34.3	Oct 70 foam pad
		4		36.7	Oct 70 foam pad
		5		22.6	May 71 foam pad
		11		22.9	May 71 foam pad
Density (after water absorption)	D1622	53.8	(lb/ft³)	36.7††	Oct 70 foam pad
		50.3		34.3††	Oct 70 foam pad
		45.6		26.3††	Oct 70 foamed in box
		28.8		21.3††	May 71 foam pad
		34.6		22.6††	May 71 foam pad
		31.2		22.9††	May 71 foam pad

*5% LP-3
†2½% LP-3
**Paired samples (cut from same piece)
††Before testing
***After testing
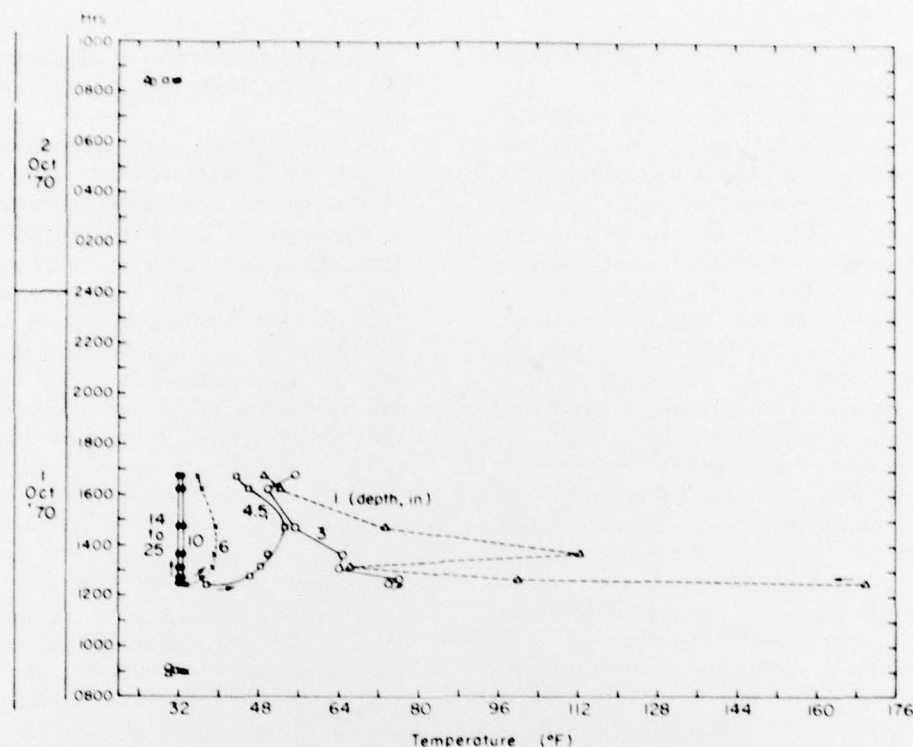†††Paired samples—cycled between 25°F and 72°F

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Figure 24. Cooling curves of section d, thermocouple assembly C.

1600 No reading   Thin foam added over most
1645 Reading   of the pad

2 October 1970

0825 Final reading

The period from 1645 hours on 1 October until the next morning is believed to be suitable for such a calculation. Although the first and last readings are not joined graphically with the rest of the curves, it can be assumed that the early set of readings joins the graphs via straight lines until the time of foaming, and that the last set of readings joins the graph by smooth curves of increasing negative slope. This can be justified by the fact that the air temperature during the period under consideration remained nearly constant.

A thermal conductivity value of approximately 0.5 Btu in./hr ft² °F was obtained by this method for the foam with an average density of 35 lb/ft³. Although the procedure is not rigorously exact, the value agrees quite favorably with an extrapolation of SWRI's results.[3]

Two California Bearing Ratio (CBR) tests were run on the front half of the October 1970 pad (Table IV). These were performed directly on the foam surface with the standard CBR equipment attached to the bucket of a small track-mounted front end loader. Both sites showed circumferential cracking during loading, indicating that the foam had failed in flexure due to the highly flexible underlying vegetative mat.

LAB TESTS FOR PHYSICAL PROPERTIES

Table IV contains all the lab test results for the various samples of sulfur foam tested. All density measurements were made on 2½- × 2½ × 4-in. samples.

There were three distinct sources of the foam samples; the most important for the purposes of this report were those from the two test sections themselves. Samples were taken directly from the pads at various times after the foam was poured on the ground and had solidified. A second source was one of the October 1970 batches from which poured samples were taken in three wooden boxes. The boxes were then packaged and shipped to CRREL in Hanover, New Hampshire. The third source was the SWRI laboratory

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at San Antonio, Texas, from which samples were shipped to CRREL for testing.

It is interesting to note the difference in densities and compressive strengths between the October 1970 foam poured in several layers on the ground and that poured in a single layer into wooden boxes at the same ambient air temperature. The foam poured into the boxes had a density range of 12.8-29.9 lb/ft³ and averaged 21.4 lb/ft³. The density of the foam pad, however, ranged from 30.0 to 38.2 lb/ft³ with an average value of 36.1 lb/ft³. The presence of moisture on the pour surface is suspected to have contributed to the higher densities in the pad.

Variations in the foams were studied in small samples (less than 2-in. cubical shapes) selected for their homogeneity. In the boxed foam, samples as light as 10.7 and 15.4 lb/ft³ were found; however, there were many dense spots and layers in the foam, accounting for the higher average density mentioned above.

A similar check was made on the foam pad containing LP-3 poured in May 1971. Its average density was 21.6 lb/ft³ with a density range of 17.6 to 26.1 lb/ft³. Some small samples were selected, as previously, and densities in the 9- to 14.0-lb/ft³ range were found. The density figures alone indicate the large variation that also occurred in this batch of sulfur foam prepared in Alaska, although better low density control is apparent for the May 1971 foam than for the October 1970 foam.

The compressive strengths of these foams were determined in accordance with ASTM test no. D1621 using the load at a deformation of 10% of the original sample height or at a lesser deformation if the maximum load occurred before 10% deformation. The majority of the samples tested reached a maximum strength value before 10% deformation occurred. Lower compressive strengths, for comparable densities, were obtained for the samples with strengths calculated at 10% deformation. These lower strengths were generally indicative of the heterogeneity of the foam.

The compressive strengths of the majority of samples tested were in the 23 to 63 lbf/in.² range for 19- to 25-lb/ft³ foam made in the boxes in October 1970. This range is for the densest concentration of the data, and a general interpretation is that this type of foam with densities of 19 and 25 lbf/in.³ has compressive strengths of 23 to 29 lbf/in.² and 56 to 70 lbf/in.², respectively. A strength vs density plot of all the test data produced no definitive relationship due to extreme scatter.

A similar scatter of the compressive strength and density data was noted for the LP-3 formulation of May 1971. This foam had a concentration of densities in the 19- to 22-lb/ft³ range with strengths in the 32- to 51-lbf/in.² range. Fewer compressive strength tests were run on pad samples than box samples from the October 1970 pour, but those run on samples in the 34 to 38 lb/ft³ range gave compressive strengths ranging from 90 to 145 lbf/in.². Representative test data for all samples are presented in Table IV.

The water absorption tests indicate that the LP-3 formulation is less absorbent than the original formulation. Field observations of the October 1970 pad indicate that it absorbed a large amount of spring meltwater (during May 1971) which contributed to the poor performance of the pad under little traffic use. Compressive strength measurements performed after the water aborption tests indicate a marked reduction in strength in the original formulation, but little loss of strength in the LP-3 formulation.

A similar strength reduction occurred after freeze-thaw tests. The LP-3 formulation lasted through more cycles, on the average, than the original formulation. All samples spalled to a small extent and typically failed from thermally induced stresses. Generally, the October 1970 foam had a substantial reduction in compressive strength after the test, while the May 1971 foam retained most of its strength.

These physical property tests were conducted over a period of 16 months after foaming the October 1970 original formulation, and nine months after the May 1971 LP-3 foaming. Samples that were brought to the laboratory after each field trip, along with those obtained from the pads at later dates, were stored in an insulated room at temperatures of 40° to 70°F before testing. No trend in the loss of strength could be ascertained over this period, except in the case of samples from the original pads that were affected by water absorption. Strength loss due to initial creep, pointed out by Dale,[2] would have subsided by the time the first samples were tested and the large variance in foam characteristics could easily mask a trend of this sort.

SULFUR FOAM ODOR ANALYSIS

The chemical reaction in the production of the sulfur foam developed by SWRI evolves hydrogen sulfide (H_2S) gas which acts as the expanding agent. Hydrogen sulfide is a toxic gas and handling precautions must be taken during the foaming operation, although the residual gas in the foam retains a questionable toxicity level. The foam produced in Alaska in October 1970 continued to have the very disagreeable odor of H_2S after 19 months. It is reported by the SWRI that lower density foams made in San Antonio, previous to the Alaskan tests, exhibited low levels of H_2S emission several days after foaming. Since the gas is necessarily present during the production of all sulfur foam and seems to diminish with foam age, an exchange of cell gas with ambient air must occur, possibly at a variable rate depending on density. Any cracking of the foam cells releases H_2S and when the foam is wet, as at the site in Alaska, the intensity of the H_2S emission seems to be greatest.

During the strength testing of the foam samples containing LP-3, it was noted that the H_2S odor was absent or possibly masked by a new odor akin to the polysulfide polymer used as the plasticizer. Consequently, a number of qualitative chemical analyses on various foam samples containing LP-3 were conducted during the period from April 1971 to March 1972. For the original samples, only the presence or absence of H_2S gas was determined. More complete analyses were subsequently made on other samples with variable results; some samples contained H_2S and others did not. A Perkin-Elmer 900 Gas Chromatograph-Mass Spectrometer system was used for these analyses.[*]

During April 1971 the first foam sample containing 2½% LP-3 (made at SWRI) was analyzed at 25°C after a one hour equilibrium period of the prepared sample. No detectable level of H_2S was determined in the sample by either the TIC (total ion current) chromatogram or the spectrum.

In November 1971 another sample was tested for the presence of H_2S. This sample had been stored at CRREL since its removal in June 1971 from the May 1971 sulfur foam pad containing 1½% LP-3. The sample was analyzed at temperatures of 25°C and 75° to 80°C. A detectable amount of H_2S was found in the sample at 25°C and a very large amount was measured in the sample at 75° to 80°C; however, the latter was attributed to the breakdown of compounds of higher molecular weight during heating.

During the storage of this particular sample some high H_2S-content sulfur foam had inadvertently been stored nearby. In order to establish whether or not the sample had absorbed a significant amount of H_2S, two new samples were obtained in December 1971. One was obtained from the pad foamed in May 1971 in Fairbanks; the other, received from SWRI, was recently made with a 2½% LP-3 content. The purpose of this analysis, made during March 1972, was to determine whether H_2S was present in the two samples and to identify any compounds which might be contributing to the odor.

Both of these samples were analyzed at 77°F in the same manner as previously used. The first (the Alaskan sample) showed a very low level of H_2S, but a very high level of an unknown compound with a base peak (highest peak) at molecular weight (m.w.) 106 of (Fig. 25). The second sample (the more recently foamed one) showed a much higher level of H_2S, some SO_2, and the same unknown compound with the base peak at m.w. 106. This new Alaskan sample contained a small amount of moisture which did not seem to have any effect on the H_2S level.

A plausible explanation for the different levels of H_2S measured in the various samples can be attributed to the method of formation of the foam. Each sample was taken from a separate mixture or batch, and consequently each sample was separate and distinct from all others, with the amounts of H_2S varying in each batch. This would account for the fact that H_2S was not detected in the first sample analyzed in April of 1971. In the most recent analysis, the older of the two samples was shown to have very little H_2S while the other sample had much more.

The unknown compound with the base or highest peak of m.w. 106 seems to be a result of the breakdown of the m.w. 1000 polymer, Thiokol LP-3 ($-S_xCH_2CH_2OCH_2OCH_2CH_2S_x-$, $x = 4$) used in the formulation. Its mass spectrum (Fig. 25) indicates that the unknown compound is probably either $C_3H_6S_2$ or $C_3H_6SO_2$. It is thought that $C_3H_6S_2$, somewhat in the form of $HS-CH_2-S-CH-CH_2$, is the more likely compound, but the $C_3H_6SO_2$ form cannot be ruled out. Both compounds result from a breakdown of the polymer LP-3. Compounds of lesser amounts also exist but these seem to be isomers of the unknown m.w. 106 compound.
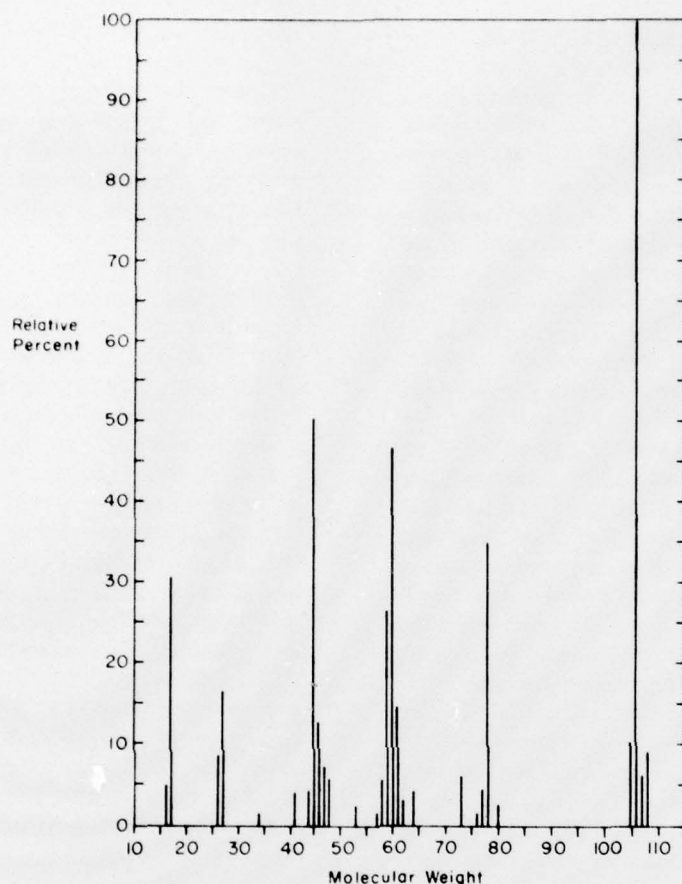
Figure 25. Mass spectra for unknown gas of molecular weight 106.

The toxicity of the unidentified compound of m.w. 106 is unknown at present. Some nauseating odors were produced in closed quarters by the last sample obtained from the SWRI; an analysis showed that the sample also contained a significant level of H_2S. An effort should be made to accurately identify the m.w. 106 compound and determine its toxicity if LP-3 is to be used in the foam formulation.

INSULATION COSTS

Raw material costs for sulfur foam are published in the final report[1] written by SWRI on the associated research contract. These costs are based on the production of a 15-lb/ft³ foam. The October 1970 foam produced in Alaska had an average density of 35 lb/ft³. The cost, therefore, for a similarly produced foam would be larger by a factor of $2\frac{1}{3}$. In addition, the relative cost of this foam would be increased by a factor of $1\frac{1}{2}$ when the poorer thermal protection of this 35-lb/ft³ foam is considered. A linear extrapolation of a graph in SWRI's report[1] yields a K-factor of 0.54 for 35-lb/ft³ sulfur foam. This value was also approximated from the cooling curves of sulfur foam shown in Figures 23 and 24. Thus the cost per 10-ft square (100 ft²), as shown in Table II of the SWRI report, would be greater than that for the other foams because of the greater quantity required for thermal protection. Even if the foam could be produced at $.016/lb, as suggested in the SWRI report by using less expensive ingredients, the lack of density control in the field during batch application would result in the sulfur foam material costing nearly as much as the polystyrene.

The addition of the Thiokol LP-3 plasticizer, to increase the foam's flexural strength at a cost of

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Table V. Cost data for varying amounts of LP-3.

LP-3 additive (%)	Parts by weight (lb)	Cost ($)	Material cost ($/lb)	Material cost ($/ft³)	Material cost ($/square)*
0	121.25	3.68	0.0303	0.455	10.50
1	122.25	4.48	0.0367	0.540	12.59
1.5	122.75	4.86	0.0396	0.594	13.85
1.85	123.10	5.16	0.0419	0.628	14.67
2	123.25	5.28	0.0428	0.643	15.00
3	124.25	6.08	0.0489	0.730	17.05

*Square = 100 ft².

Note: Cost data are for 1972.

$0.80/lb in the May 1971 test, is relatively expensive. Referring again to the relative costs of the foams in SWRI's final report, the addition of LP-3 in various percentages to increase flexural strength with no reduction in the compressive strength or thermal protection would result in the costs shown in Table V.

LP-3 additive exceeding 1.85% by weight gives the sulfur foam a greater cost per square (100 ft²) than that of polyurethane (@ $14.70), and the cost of polystyrene is exceeded with less than 1% LP-3. The May 1971 foam installed in Alaska had a minimum of 1½% LP-3. In addition, the 21.6-lb/ft³ average density of the installed foam would raise the relative sulfur cost above that indicated by the SWRI for 15-lbf/ft² foam.

LAB EXPERIMENTS WITH SULFUR FOAM

During the course of the sulfur foam contract with SWRI, a limited lab program at CRREL involving sulfur foam experimentation was initiated. This program was deemed necessary to gain some first-hand experience with the mechanism involved in the production of foamed sulfur. Such information would give a better understanding of the foaming process, which hopefully would lead to simplification and possible improvement in the production process and the foam physical properties. Two objectives received the most attention. The first was to determine which gases, and what quantities of each, would dissolve in molten sulfur. The second objective was to investigate the possibility of making simplifications in the original formulation or finding more desirable components to foam the sulfur.

The replacement of H_2S, with its toxicity and stench, by some other blowing agent was a high priority objective of these studies. The simplest approach was to attempt to find which gases (or liquids in the gaseous state) would dissolve in molten sulfur at temperatures greater than 112°C. These compounds would be dissolved in the molten sulfur under pressure and would expand the mixture upon exposure to atmospheric pressure. The internal reactor pressure necessary to expel the liquid would confine the gas within the liquid sulfur until expulsion occurred. Such a gas substitution would eliminate the need for a chemical reaction to produce the blowing agent. The production of a less toxic gas than H_2S in the sulfur mixture from an additional or different chemical reaction is possible, but would probably complicate the formulation further. Likewise, the mechanical addition of a blowing agent by high-speed mixing has the drawback of requiring additives to increase bubble film strength. Consequently, emphasis was placed on dissolving a gas in liquid sulfur.

A 1000-ml pressure vessel (Parr Instrument Co. Model 4511) was used for the solubility tests and for conducting some of the simpler chemical reactions. Considerable modification of the vessel was required for injecting gases and liquids and for the expulsion of the sulfur foam. A 600-g charge of sulfur was used in the majority of the experiments, leaving 665 ml of free space for pressurizing. All solubility tests were made in liquid elemental sulfur in the temperature range of 120° to 172°C, most being conducted at 140°C. The following significant observations were made:

1. Air and carbon dioxide (CO_2) seem completely insoluble without an additive.

2. Freon 12 (CCl_2F_2), Freon C-318 (C_4F_8), and sulfur hexafluoride (SF_6) appear to have little if any solubility.

3. Carbon tetrachloride (CCl_4), chloroform ($CHCl_3$), methylene chloride (CH_2Cl_2), and methyl chloride (CH_3Cl) all have more solubility.

4. No chemical reaction occurred between the preceding substances and the sulfur.

5. Sulfur will react with simple hydrocarbons to produce H_2S.

Table VI lists the tested substances that have some solubility in liquid sulfur plus a number of others which could be substituted for the present H_2S blowing agent. The absolute solubility of these substances in molten sulfur is unknown at present, as is their solubility in the reacted

Table VI. Relative amounts of potential sulfur foaming agents required to produce a 12-lbf/ft² foam.*

Substance	(lb)	Liquid density (g cm³)	Boil point at 1 atmos (°C)	Molecular weight	Heat of vaporization†
CCl₄	3.4	1.59	76.5	154	46.4
CHCl₃	2.6	1.48	61.7	119	59.0
CH₂Cl₂	1.9	1.33	40	85	78.6
CH₃Cl	1.1	gas	−24	50.5	
Freon 11	3.0	1.47	23.8	137	43.0
Freon 12	2.7	gas	−29.8	121	39.5
Freon 13	2.3	gas	−81	104	35.5
Freon 21	2.3	1.37	8.9	103	57.9
Freon 22	1.8	gas	−40.8	80	55.8
Freon 114	3.8	1.46	3.8	171	32.5
Freon 500**	(3)	gas	−33.5	**	
Freon 502††	(2.5)	gas	−45.6	††	42.5

*Contained in a kettle of molten sulfur under pressure. 10 ft³ of gas in 100 lb of sulfur at 112°C and 1 atmosphere.
†Btu/lb °F
**Azeotrope. 74 wt % Freon 12, 26% Freon 152.
††Azeotrope: 49 wt % Freon 22, 51% Freon 115.

polymeric mixture. The table also shows the minimum amount of each gas, if full expansion at 112°C is realized, that must be contained within the foam to produce a 12-lb/ft³ foam.

The second objective of the lab program — investigating the possibilities of simplifying the standard SWRI foam formulation — was prompted by some skepticism about the necessity of reacting five ingredients with sulfur to produce the foam. If sulfur foam could be produced from fewer ingredients, product uniformity would probably be easier to achieve.

Another aspect of this second objective was the possibility of finding more desirable components than some of those currently used. Since no change in formulation was immediately evident, basic experimentation was conducted to characterize the foam process.

The interaction of 1,5-cyclooctadiene with phosphorus pentasulfide (P_2S_5) and the molten sulfur is apparently a polymerization process which produces, plasticizes and strengthens the resinous substance of undefined chemical composition which is the basis of the foam. The reaction of phosphoric acid with this substance evolves hydrogen sulfide which expands the mixture into a foam.

Experiments indicate that squalene, a component of shark liver oil, is a possible replacement for the 1,5-cyclooctadiene, although the reaction product of squalene and P_2S_5 probably

has limited solubility in liquid sulfur. Other substances, such as nylon, polyethylene and polystyrene, were tried as possible plasticizing agents, but results were negative because of their insolubility in liquid sulfur. Ditert-nonyl polysulfide forms an emulsion in liquid sulfur and appears to dissolve a sizable amount. This yellow liquid is a possible substitute for the tricresyl phosphate.

Inefficient laboratory ducting of odors released from some of the chemicals used in these experiments caused an extended postponement of further experimental work. At that time the following work was tentatively planned:

1. Additional tests of foaming agents to include quantitative analyses of their solubility in liquid sulfur.

2. Continued partial reactions of SWRI's foam components to find likely chemical substitutions for simplifications of their formulation.

3. Test tube evaluations of chemically modified 1,5-cyclooctadiene and squalene as toughening agents, and subsequently utilizing these products to produce a foam.

CONCLUSIONS

The sulfur foam did not function satisfactorily as an expedient road insulation because of its low tensile strength and high brittleness.

The batch process of foam production results in highly heterogeneous foam with respect to density, especially if moisture is present on the substrate surface when pouring the foam.

Water absorption is an important consideration deserving attention, and waterproof coatings should be investigated.

The insulating value of the field batch sulfur foam is about one-half that of extruded polystyrene and with any water absorption it becomes even worse.

The H_2S foaming agent* is highly undesirable and probably prohibits the use of the foam without an impermeable surface coating in building construction.

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Research conducted by the Chevron Research Company since these tests has successfully replaced the H_2S with CO_2 as the blowing agent.

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    v, 26 p., illus.; 27 cm. (CRREL Report 79-18.)
    Prepared for Directorate of Military Programs -
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I. Pazsint, Daniel A., joint author. II. United States.
Army. Corps of Engineers. III. Army Cold Regions Re-
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IV. Series: CRREL Report 79-18.